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THE TENSILE AND CREEP PROPERTIES OF TITANIUM, SOME BINARY TITANIUM ALLOYS, AND SOME TERNARY LLOYS BASED ON A TITANIUM 5 % ALUMINIUM ALLOY

REPORT ON WORK CARRIED OUT BRITISH NON-FERROUS METALS RESEARCH ASSOCIATION UNDER AGREEMENT

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THE TENSILE AND CREEP PROPERTIES OF TITANIUM, SOME BINARY TITANIUM ALUMINIUM ALLOYS, AND SOME TERNARY ALLOYS BASED ON A TITANIUM 5% ALUMINIUM ALLOY

by

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With APPENDIX

by

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Report on work carried out by The British Non-Ferrous Metals Research Association under agreement No.7/Exptl/527/R3 for the Ministry of Supply.

SUMMARY

This report describes a study of the tensile and creep properties of titanium, titanium-aluminium binary alloys, and of ternary alloys containing as major constituents in addition to aluminium, boron, carbon, chromium, columbium, iron, molybdenum, manganese, silicon, vanadium, tungsten or zirconium.

As in earlier work described in another report (1) the object has been to find alloys with useful creep properties at temperatures up to 650°C., but in the work to date the highest test temperature has been 500°C. In the earlier work titanium-base alloys were melted and cast in graphite, and then rolled to strip for mechanical tests, but in the present work an arcmelting technique was adopted to avoid contamination of the alloys with carbon.

In the earlier work a wide range of binary alloys was tested and it appeared that alloys containing aluminium were particularly promising. Unlike the other metallic alloying elements used, aluminium raises the alpha/beta transformation temperature(2) in titanium, and tests described here show that titanium-aluminium alloys can only be hot worked easily at temperatures near to or in the alpha plus beta or beta phase fields. Bearing in mind the potential applications of any alloys with useful properties at elevated temperatures it appeared desirable to restrict the hot working temperature to not more than 1000°C., and partly for this reason the ternary alloys described in this present work were based on titanium 5% aluminium alloys, although a somewhat higher aluminium content would be compatible with this requirement. A further desirable requirement was that the materials should have not less than 5% elongation in tensile tests; at the outset of the work it appeared that on this score also the aluminium content would have to be restricted to 5% although, as the results in this report show, alloys of somewhat higher aluminium contents also gave the necessary ductility.

The binary and ternary alloys were hot-rolled to strip and if the hot-rolled materials had elongations of 5% or more they were tested in that condition. Less ductile materials were annealed at 1,000°C. and slowly cooled in an attempt to improve their ductility, and where this was successful the annealed materials were also tested.

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The structures of the materials were studied by metallographic and X-ray methods, and the report includes some account of the complex structures observed and of the way in which these structures arose while the metallography of the materials examined is described in detail in an Appendix. The titanium-Aluminium alloys had all alpha structures, whose appearance under the microscope differed markedly, depending upon the conditions under which alpha was formed from the beta phase existing at hot working or annealing temperatures. Most of the ternary alloying additions that were used were beta stabilisers and again a variety of micro-structures were observed depending upon the conditions under which the alpha plus beta structures were formed. In certain cases, notably in alloys containing chromium or iron, slow eutectoidal decomposition of the beta phase during prolonged exposure to temperatures of the order of 400°C. and 500°C. caused severe embrittlement.

The tensile properties of the alloys ranged from about 50 tons per square inch U.T.S. and 18% elongation for the annealed titanium-5% aluminium alloy to figures of the order of 90 tons per square inch and 3% elongation for some of the highly alloyed ternary alloys. In general the highest strength compatible with a minimum ductility of 5% elongation was about 75 tons per square inch although in a number of individual cases higher values of the order of 85 tons per square inch, were recorded.

The properties of the materials at elevated temperatures were explored by stress-rupture tests at 400°C. and 500°C. The tests were of 500 hours duration and were designed to reveal the stress producing 1% or less extension in that time.

For the hot-rolled titanium at 400° and 500°C. these stresses are approximately 10 and 2 tons per square inch respectively. For the hot-rolled titanium 5% aluminium alloy they are about 30 and less than 12 tons per square inch respectively, while for the 8% aluminium alloy they are 35 and not less than 14 tons per square inch. Some of the ternary alloys have considerably better creep properties, and alloys containing 5% or 10% molybdenum or 1% silicon or 10% zirconium will withstand stresses of the order of 38 tons per square inch at 400°C. and extend less than 1% in 500 hours. At 500°C. the ternary alloys containing 5% aluminium are not much better than the binary 8% aluminium alloy.

It is likely that the creep properties of the materials are dependent in part on their grain size. No evidence on this point is available in the present work but the grain sizes of the materials have been recorded and it is worthy of note that the binary 5% aluminium alloy was coarser grained (~0.03 mm. grain dia.) than the 8% alloy and most of the ternary alloys (~0.003 mm.).

The binary and ternary alloys were quenched from 1000°C. and aged at 400° and 500°C. to determine whether their mechanical properties could be usefully improved by such treatments. The alloys containing chromium, iron, manganese and vanadium all hardened markedly and in the cases where tensile tests were carried out on unbroken stress-rupture test pieces (chromium and iron alloys) the hardening was associated with severe embrittlement. It seems unlikely that heat treatments of this kind could be applied profitably to these alloys.

Of the ternary alloys with promising creep properties only those containing zirconium tended to be embrittled by exposure at elevated temperatures, and in these cases only at 500°C. The tensile properties of the alloys containing molybdenum or silicon were not adversely affected by such heat treatments.

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PREPARATION OF MATERIALS

Materials

The compositions of the titanium metal used for the preparation of the alloys are set out below, most of the materials being made from the sponge Q.A.B., which after arc melting gave hardness values averaging 180 V.P.N.

		Composition %	
	Iodide Ti	Sponge (Q. A. B.)	Sponge (OON)
С	0.02 - 0.03	0.06 Ø	
Cl	-	0.06 \$	0.05 - 0.10
Mg	0,005	0.03 \$	0.01 - 0.05
Fe	0.01 - 0.05	0.05 Ø	0,05
02	0.031 [±]	0,085 °	0.07 0.
N ₂	0,05 ^H	0.05 °	0.01 - 0.03

Suppliers analysis figures

The remaining values are from a paper by Gee et al (3) and are for unmelted titanium.

Super-pure aluminium was used and the silicon was from a specially purified batch. The remaining addition elements were of high commercial purity.

Composition of Alloys

The nominal or actual compositions of the alloys are included in the tables of results to which reference is made later. Oxygen analysis was carried out with the Association's vacuum fusion apparatus (5).

Arc Melting

The furnace has been fully described elsewhere (4). Melting takes place in a water-cooled copper mould, the base of which is retractable, and the arc is struck between the metal to be melted and the -Ve tungsten electrode. The alloys were melted in 40 cm. Hg. pressure of argon, purified by gettering with molten titanium. Most of the ingots were 2 in. x 1½ in. x 1 in. in size, weighed 200 gms. and were made up from ten 20 gm. lots. The sponge titanium for each lot was pressed into a wafer approximately 1½ in. x 1 in. x ¼ in. and then baked out at about 1400°C. in vacuo, to remove residual magnesium chloride which otherwise caused considerable sputtering when the sponge was melted. The appropriate amount of addition element was then pressed into the titanium compact; additions which were in powder form were enclosed between two leaves of aluminium foil and pressed in as capsules. Each compact was melted into a button which was remelted twice, being turned over each time; without this premelting of the compacts some segregation was found in the hot rolled products.

The premelted buttons were then built up into an ingot by introducing them one at a time into the melt and using power sufficient to maintain at least twice the volume of one button molten.

^{*} B.N.F. analysis figures

O B.N.F. analysis figures on metal worked down to sheet.

Analysis figures given in the tables show that approximately 0.02% tungsten was picked up during melting.

Hot Working

Earlier observations had indicated that titanium aluminium alloys required comparatively high working temperatures. In the present work, preliminary hot rolling tests with 20 gm. buttons of alloys containing up to 40% aluminium, made from the sponge OON by arc melting were carried out. These gave the results represented by Fig. 1 which also includes data obtained from the rolling of titanium-aluminium alloys melted in the Association's graphite resistance furnace. In these tests the rolls were heated to about 100 to 150°C., and 15% reduction was imposed in a single pass. The figure shows that as the aluminium content of the alloys increased it was necessary to raise the rolling temperatures to avoid cracking.

The ductility of a narrower range of titanium-aluminium alloys was examined in more detail by impact tensile tests. The speed of deformation in these tests is somewhat faster than in hot rolling, but approximates more to the rate of deformation in rolling than to that in ordinary tensile testing. The results are detailed in Table I, from which it can be seen that there is a sudden increase in the ductility of the alloys at a temperature depending on the aluminium content. Reference to the phase diagram shows that this occurs when the alloys enter the alpha plus beta or beta phase fields. The results of these tests are described in more detail later, but it will be noted from the Table that alloys containing more than 5% aluminium do not develop high ductility at a temperature of 1000°C., the temperature adopted in the present work for fabrication of the binary 5% aluminium alloy and the ternary alloys based on it.

Except where otherwise stated, all of the alloys described here were pre-heated to 1000°C. in air, and rolled in air, with the rolls preheated to 100°C. - 150°C., until the thickness of the ingot was reduced from approximately 1 in. to 0.25 in. In most cases the furnace temperature was then lowered to 950°C. and the sheet reduced to 0.060 in., if required. The ingots were cross-rolled twice in the early stages of rolling, and were reheated between each pass of approximately 20% reduction.

At 1000°C. all the ternary alloys based on titanium 5% aluminium had reasonable hot working characteristics, but some edge cracking took place in those alloys containing boron, carbon, silicon, tungsten and vanadium additions. In no case was this severe enough for the sheet to be scrapped and, at its worst, cracks up to ½ in. long, running into the sheet, occurred in strip 3 in. wide. The cracking was generally worse at the higher alloy contents and vanadium additions had the greatest detrimental effect.

The maximum aluminium content of a binary alloy which could be hot worked at 1000°C., was 8% (using sponge Ti Q.A.B.). At this composition edge cracking occurred when an attempt was made to lower the working temperatures as the sheet became thinner.

Machining

It has been found convenient to cut sheet material with a slitting wheel, although, if cracking is to be avoided, care is necessary with some of the alloys, notably those containing chromium or iron additions. This difficulty was mentioned in an earlier report(1) and a method of avoiding it given. The rate of wheel wear for slitting and grinding titanium and its alloys is considerably greater than for other more usual metals.

For other machining operations the best results have been obtained with carbide-tipped tools using slow cutting speeds. Some unalloyed titanium billets have been cut with a power hacksaw. Slow heavy cuts gave the best results and kept blade wear to a minimum.

Contamination with Oxygen and Nitrogen in Hot Working

The surface layer formed on the hot worked titanium and titanium alloys was removed by the use of two acid pickles in place of the sodium hydride bath (6) and acid pickle previously used (1). The first bath was a 15% hydrochloric acid mixture in which the specimens were allowed to soak for periods of up to 30 minutes depending on the composition of the alloy. This effectively loosened the layer and enabled most of it to be scrubbed off. The tenacity of the layer increased as the aluminium content of the alloy increased and was greatest on the alloys containing 8% aluminium or more. The second bath was a nitric-hydrofluoric acid mixture which removed the last traces of surface contamination and left a "bright-pickle" surface finish.

Table VII gives the hardness of titanium and the alloys in various conditions and it can be seen from the figures that any contamination which occurred on hot working in air at 1000°C. did not cause hardening of these materials. It may be inferred from this that if any oxygen or nitrogen was absorbed during this fabrication, the effect was confined to the surface layers.

This was confirmed for oxygen, by taking a piece of 0.050 in. thick titanium, hot rolled at 1000°C. and turning off different thicknesses of surface layers. These layers were turned off from both sides of the sheet and at any one part of the sheet the same depth was taken off from both sides. Oxygen analyses were done on the different thickness of remaining metal.

The results of these analyses and the thicknesses of the samples are given below:-

Thickness	Depth turned	Oxygen
of remaining	off from	Content
metal	surface	%
0.050 in.	nil	0.484
0.045 in.	0.0025 in.	0.097
0.035 in.	0.0075 in.	0.084
0.025 in.	0.0125 in.	0.086

The pickling process described above removed a surface layer about 0.004 to 0.008 inch thick and the results above indicate that this eliminated contamination with oxygen arising from the hot-working operations.

METALLOGRAPHY OF THE MATERIALS

The following brief account of the metallography of titanium and its alloys will help the reader to recognise various structural features of the materials referred to in later sections of the report.

A fuller account of the metallography of the materials is given in the Appendix.

Great care is necessary in the interpretation of the titanium alloy structures as revealed by the microscope and the aid of X-rays is almost always necessary. The main reason for this is that titanium itself undergoes a transformation during cooling from cubic body-centred beta titanium above 885°C. to hexagonal close-packed alpha titanium below this temperature. The transformation is affected differently by various alloying elements and the resultant structures naturally differ according to rate of cooling and heat-treatment. In addition, certain alloy systems require very long times to attain equilibrium at temperatures below about 800°C., and this must also be taken into account.

As an illustration of the diversity of structure which can be exhibited by a single phase, Plate I shows four entirely alpha structures. 'A' was formed by quenching from the beta region and is a typical transformed structure. 'B' shows a dendritic structure formed on slow cooling through the transformation point, followed by annealing at a lower temperature. Similar structures are formed when quenched material is annealed in the alpha range provided no recrystallisation takes place. 'C' shows equiaxial alpha grains formed on recrystallisation in the alpha range, and 'D' an apparently two-phase structure formed on quenching from the alpha+ beta range, the two components being in fact alpha which has been formed rapidly from beta, and alpha which was stable at the quenching temperature.

A great variety of alpha-beta structures was obtained. In some, fine equiaxial alpha grains were connected by a grain boundary film of beta, (see Plate IIA). In others, a very coarse grain size at the quenching temperature results in very large grains consisting of platelets of alpha and beta laid down in geometrical array; an example of this structure is shown in Plate IIB. Again, small particles of alpha might be distributed in the grains and along the boundaries, the matrix consisting of beta and alpha (Plate IIC) or alpha might appear as spines in a beta matrix (Plate IID).

The only all-beta alloy studied gave a similar appearance to the all-alpha alloy illustrated by Plate IC.

It can be seen from the above that, whereas an alloy consisting of beta only cannot show a transformed structure, alpha can take almost any form, and alpha+beta structures can vary widely.

A further characteristic of these alloys is that material containing alpha and beta at temperatures below the transformation range can be converted to either all alpha or all beta, depending on the composition. Similarly the proportion of the two constituents may be altered in either direction. The behaviour in any system depends on the beta-stabilising characteristics of the alloying elements (11). The way these different changes can occur is explained below.

Suppose Fig. 2 represents the relevant part of the equilibrium diagram of titanium containing 5% of aluminium with a third element M. There exists for certain alloy systems a content x5 of M, above which the beta phase is retained on quenching from the beta field and below which the beta is transformed to alpha. Consider now an alloy containing a% of M, quenched from the beta field. All the beta is transformed to alpha. When cooling from the beta field is slow, the first alpha to separate has composition b and the remaining beta composition c. According to the usual rules, these compositions change along the lines bd and ce while cooling continues, until the last alpha separating has composition d and the last beta to transform to alpha has composition e. The resultant structure consists entirely of alpha. But now suppose we take an alloy containing m% of M. On quenching from the beta field the alloy is all alpha as before, but on slowly cooling from the beta field, or on quenching from lower temperatures a different result ensues; for, when the temperature represented by the line vn is reached, alpha of composition v forms and the composition of the residual beta changes, during further cooling, along the line nrt. At the temperature represented by qr, the residual beta becomes stable, and no longer transforms on quenching. Alloys quenched from below this temperature therefore contain beta in quantities decreasing as the temperature of quenching decreases, although quenching from the beta field gave entirely alpha structures. Compositions lying to the right of xx consist of beta on quenching from the beta field and alpha+beta on quenching from the alpha+beta field.

Bearing in mind the features described above, it becomes possible to interpret logically the structures of all the alloys examined in the present survey.

IMPACT TENSILE PROPERTIES OF THE TITANIUM ALLOYS

Specimens of dumbell shape with 0.225 in. diameter by ½ in. long gauge lengths machined from ¼ in. thick hot rolled plates were used for impact tensile tests. The specimens were preheated in a furnace close to the Izod machine and kept at the testing temperature for about three minutes. About 2 seconds elapsed from the time the specimen was taken out of the furnace to the time it was broken. The energy of the swing was 120 ft. lbs. for all the room temperature tests and 40 ft. lbs. for the elevated temperature tests. One half of each specimen was water quenched within one second of fracture.

Binary titanium alloys containing 5%, 8% and 10% aluminium were tested at room and elevated temperatures, mainly to obtain a measure of their ductility at the temperatures at which the materials might be forged or otherwise hot worked. Two 5% aluminium ingots were tested, one made up with sponge titanium (Q.A.B.) and the other with iodide titanium (QAC). This latter ingot was, however, made up before the present melting technique was established and microexamination indicated that there was some segregation across the section of the rolled material. The remaining ingots were made up using titanium sponge (QAB) by the improved premelting method and were apparently free from segregation.

The 5% and 8% aluminium alloys were hot rolled at 1100°C. and the 10% aluminium alloy at 1180°C. These materials were quenched after the last pass through the rolls. They all had transformed structures of the type shown in Plate IIIA showing that the metal was in the beta phase field at the rolling temperature.

Examination of the quenched part of the impact tensile specimens showed that at the lower testing temperatures this transformed structure persisted during the period that the specimens were held in the alpha phase field prior to testing.

With increasing temperature some changes in structure appeared particularly at temperatures above the alpha/alpha+beta transus and some heat treatments (described in the Appendix) were carried out on the rolled materials to determine the nature of the changes.

The result of these tests (Table I) indicate that alloys with up to at least 10% aluminium have fair elongation at room temperature.

For all the alloys tested the ductility increases rapidly at temperatures corresponding to the alpha/alpha+beta boundary of the binary phase diagram. Microexamination of the quenched test pieces confirmed that the materials were in the alpha+beta or beta phase fields when they displayed high ductilities. These results were consistent with those described earlier in this report on the rolling trials of the titanium-aluminium binary alloys.

The 5% aluminium alloy (PZX41) made from iodide titanium was slightly more ductile but weaker (c.f. energy to fracture values) than the corresponding alloy (PZX54) made from sponge titanium.

The structures produced in these particular alloys by rolling were clearly unstable, and the results of the tests may have been affected by changes of structure occurring when the test pieces were brought up to temperature.

TENSILE PROPERTIES

The tensile properties of the materials were determined on a Hounsfield Tensometer. Specimens were cut from blanks 3 in. long x $\frac{1}{2}$ in. wide and had a gauge length 1 in. long and $\frac{1}{4}$ in. wide.

Results are the average of two or more specimens unless otherwise stated and are given to the nearest ½ ton or ½. In most cases the results for any one material fell within the following limits:-

U.T.S. and L. of P. - ± 1 ton/sq.in.

Elongation on 1 in. - ± 1%

Reduction of area - + 3%

The limit of proportionality for these materials as determined on the Hounsfield Tensometer approximates roughly to the 0.2% proof stress.

The room temperature tensile properties of the sponge titanium and alloys based on it, in various conditions, are given in Table II. The sheet titanium made from the sponge QAB used for most of this work has a room temperature tensile strength of about 36 tons/sq.in., and an elongation of 20% in the 'as rolled', annealed, and heat treated conditions.

In the 'as rolled' condition the titanium 5% aluminium alloy (PZX 46) is stronger and less ductile than when annealed, showing that some effects of working remain in the material; at the final rolling temperature the alloy is nearly all alpha phase, but micro-sections showed that, unlike the titanium specimens, the crystals of the titanium-aluminium alloys were severely distorted and showed clear evidence of retained work.

Ternary additions to the titanium 5% aluminium alloy nearly all strengthen the 'as rolled' materials at the expense of ductility. Additions of 0.42% boron, 0.75% and 1.5% carbon, 12% chromium, 5% iron, 5% and 10% molybdenum, 2% silicon and 3.9% and 8.7% vanadium reduce the ductility to less than 5%. In the case of the 12% chromium, 5% and 10% molybdenum and 8.7% vanadium alloys, an anneal at 1000°C. restored the ductility to more than 5% elongation.

In the 'as rolled' condition the 5% aluminium 12% chromium alloy was all beta phase, (Plate IVA), but after annealing a considerable amount of alpha had been formed (Plate IVB). Annealing the 5% aluminium 5% molybdenum alloy, (see Plates IVC and IVD), and the 5% aluminium 8.7% vanadium alloy (Plates VA and B), had the effect of markedly coarsening the alpha phase particles in the beta matrix.

The alloys containing 0.42% boron, 0.75% and 1.5% carbon, 5% iron, 2% silicon and 3.9% vanadium, were brittle both in the 'as rolled' and annealed conditions. The high oxygen content of the 3.9% vanadium alloy (0.36% 02) probably accounts for its brittleness in both conditions as the alloy with a higher vanadium content (8.7%) was duetile when annealed.

The room temperature tensile properties of stress rupture specimens (see later section) which were unbroken after 500 hours, at 400°C. or 500°C., and had extended less than 1%, were determined and the results of these tests are included in Table II. The properties of the as rolled titanium 5% aluminium alpha alloy are not significantly changed by this heat treatment, suggesting that in this alloy no strain relief takes place with heat treatments of up to at least 500 hours at 500°C.

Ternary alloys containing chromium and iron were completely embrittled by this heat treatment. It would be expected that the ternary alloys containing manganese would also become embrittled by a low temperature heat treatment, as in the binary titanium systems containing chromium, iron and manganese, a cutectoid decomposition of the beta phase takes place (7, 8, 9). Plate VC and D shows the titanium 5% aluminium 5% chromium alloy in the rolled condition and after ageing at 500°C. The fine precipitate present in the aged alloy is presumably Cr3Ti2, and the embrittlement may be attributed to its dispersion in the alpha matrix.

In the remaining alloys, which were pulled after stress rupture testing, some strength improvement was produced in most cases, notably in alloys containing 10% columbium, 5% molybdenum and 10% molybdenum.

Two of these had ultimate tensile strengths greater than 80 tons/sq.in., and retained not less than 5% elongation.

The alloy containing 5% tungsten had lost its ductility after being at 400°C. for 504 hours, and alloys containing 5.1% and 9.5% zirconium lost their ductility after treatment at 500°C. but not at 400°C.

STRESS-RUPTURE PROPERTIES

Stress-rupture creep tests were carried out on creep units of B.N.F. design using the same size specimens as for the tensile tests. Loads were applied using a 5:1 lever ratio. The temperature control was \pm 1°C. at 400° C. and 500° C. and uniformity of temperature along the gauge length was controlled to \pm 1°C. The specimens were raised to temperature in 3 hours and held at temperature for 21 hours before the load was applied. After completion of 504 hours (provided that the specimen had not failed) the test was ended and the extension of the 1 in. gauge length measured with deviders. The accuracy of this method of measurement is $\pm \frac{1}{2}$.

The grain size of these materials is included in the tables giving the stress rupture time results. The effect of grain size on the results may be considerable but to date no comparative tests have been done on any alloy with different grain sizes.

The stress-rupture time properties of titanium at 400°C. and 500°C. are given in Table III and those of the alloys at 400°C. and 500°C. are given in Tables IV and V respectively. These tests were carried out for times up to 504 hours and the extensions of broken and unbroken specimens measured.

The test stresses were varied to determine the stress producing 1/2 or less extension in 504 hours.

The stresses producing 1% or less extension in the hot rolled titanium at 400°C. and 500°C. are approximately 10 and 2 tons/sq. in. respectively; for the titanium 5% aluminium alloy they are approximately 30 and less than 12 tons/sq. in.: for the titanium 8% aluminium alloy they are 35 and not less than 14 tons/sq. in. showing that aluminium additions considerably improve the elevated temperature properties of titanium.

At 400°C. the best of the ternary alloys, based on titanium 5% aluminium withstood stresses (less than 1% extension) not less than 38 tons/sq.in. (for 5% and 10% molybdenum and 1% silicon additions) and 40 tons/sq.in. (for a 10% zirconium addition).

at 500°C., these stresses are not less than 14 tons/sq.in. (for 5% molybdenum, 1% silicon, 5% tungsten and 10% zirconium additions). The best result so far obtained at 500°C. is for the 5% molybdehum ternary alloy, which withstood a load of 18 tons/sq.in. for 504 hours with 1% extension.

In general, all the alloys have better high temperature properties than titanium itself, and, having regard to the fact that these are single specimen tests, the molybdenum, silicon and zirconium alloy additions to the titanium 5% aluminium alloy give the most improved properties. The 10% zirconium and 5% molybdenum additions gave the best alloys at 400°C. and 500°C. respectively.

AGEING PROPERTIES

The effect of quenching and ageing the alloys was followed by hardness determinations. In addition, specimens in the annealed, rolled and quenched conditions were examined both metallographically and by X-rays. Tables VI and VII give respectively the X-ray results and the hardness values of the alloys in various heat treated conditions.

Small specimens about $\frac{1}{2}$ in. square were cut from hot-rolled strip and were heated in a vertical tube furnace at 1000°, 800° or 600°C. for 1 hour,

2 hours, or 5 hours respectively before quenching into a water bath.

The furnace atmosphere was argon, purified by passing it first over phosphorus pentoxide and then through titanium turnings at 1000°C. The rate of flow of argon during the heat treatment was 3 to 4 litres/hour.

Batches of quenched specimens were clamped between titanium strips and the hardness of the quenched materials was determined on the cross-section of the specimens. The batches were aged at 400°C. or 500°C. ± 5°C. in an electric muffle furnace and hardness determinations carried out at suitable intervals.

The results for titanium and some selected alloys containing 5% aluminium and 5% aluminium with boron, columbium, molybdenum, silicon, tungsten and zirconium are summarised in Table VIII. Those for alloys containing 5% aluminium with additions of chromium, iron, manganese and vanadium are given graphically in Figs. 3, 4, 5 and 6 respectively.

Neither quenching, nor subsequent ageing at 400°C. or 500°C. had any great effect on the alloys containing 5% aluminium or 5% aluminium with boron, columbium, silicon, tungsten or zirconium additions. The alloys containing 5% aluminium with 3% and 5% tungsten additions showed some hardening after ageing at 400°C.

The alloys containing 5% aluminium with 5% and 12% chromium additions both hardened appreciably on ageing at 400°C. and 500°C. (Fig. 3). Approximately the same maximum hardness values were reached at both temperatures, but the alloy containing 12% chromium reached a higher maximum value than the one containing 5% chromium. Both alloys retained this maximum at 400°C. up to at least 30 days but slowly softened at 500°C. The 5% chromium alloy softened more rapidly than the 12% chromium alloy.

The 5% aluminium 5% iron alloy hardened considerably at both temperatures, (Fig. 4), retained its hardness at 400°C. for at least 15 days and slowly softened at 500°C.

The alloy containing 1/ manganese showed no response to ageing, but the 5/ manganese alloy hardened considerably at both 400°C. and 500°C. (Fig. 5) retained its hardness at 400°C. for at least 15 days, and slowly softened at 500°C.

Additions of vanadium to a titanium 5% aluminium alloy increased the hardness of the metal considerably, particularly in the case of 5% and 10% vanadium additions. Both these alloys age hardened, in particular, the hardness of the 10% vanadium alloy aged at 400°C. (Fig. 6), was still increasing after 16 days, but slight softening occurred at 500°C.

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TABLE I

Impact Tensile Tests on Arc-Melted Titanium and Titanium Alloys

_						
	B.N.F. Mark	Composition %	Test Tempera- ture °C.	Elonga- tion %	Redn. of Area %	Ft. 1bs. absorbed
	PZY26/4 /6 /5 /2 /3 /1	Ti (Sponge Q. A. B.)	Room 400 700 800 850 900	23 45 54 50 53 66	35 25 80 80 87 100	49 ,29 17 15 13
	PZX41/3 /5 /6 /7 /8 /10 /11 /4	Ti 4.1 Al 0.08 N ₂ (Iodide Ti Q.A.C.)	Room 480 580 680 780 880 980 1080	21 35 36 36 27 34 40 85	30 70 70 65 35 70 80 100	38 31 28•5 25 17•5 17•5
	PZX54/2 /3 /5 /1 /6 /4	Ti 5.1 Al 0.14 N2 0.006 W (Sponge Q.A.B.)	Room 400 700 800 900 1000	17 26 30 33 32 63	25 30 57 63 46 80	60 39 29 24 24 21
	PZX55/1 /2 /3 /4 /5 /8 /6	Ti 8.3 Al 0.11 N2 0.025 W (Sponge Q.A.B.)	Room 400 700 900 1000 1050 1100	17 17 ≭ 32 27 30 84 99	27 25 % 46 45 45 100	60 39 31 28 26 19
	PZX56/1 /2 /3 /9 /4 /5 /8 /6	Ti 10 Al (N) 0.13 N2 0.024 W (Sponge Q.A.B.)	Room 400 700 800 900 1000 1050 1100	13 18 ³⁴ 26 24 26 23 33 85	20 32 [#] 30 30 35 30 35 100	54 33 29 30 26 23 17

⁽N) Nominal composition.

^{*} Specimen did not break

TABLE II

Room Temperature Tensile Properties of Arc-Melted Titanium and Titanium Alloys

-						
B.N.F. Mark	Composition %	Condition [™]	U.T.S tons/ sq.in.	tons/	Elonga- tion % on 1 in.	Redr of Area
PZY 23	Ti 0.094 02 0.08 N2 0.018 W	Ann. 1000°C.	38	32	18	29
PZY 31	Ti 0.116 02 0.08 N2 0.014 W	As Rolled	33.5 31	30 27	22 22.5	40 28
11		As Rolled + 606 hrs. at 400°C. As Rolled + 604 hrs. at 500°C.	35	31 29	21	42 \$
PZY 35	Ti 0.085 02 0.023 W	As Rolled	36.5	32	18.5	33
PZX 46	5 Al (N) 1.120 02 0.08 N2 0.027 W	As Rolled Ann. 1000°C. As Rolled + 504 hrs. at 400°C. As Rolled + 504 hrs. at 500°C.	63.5 50 66	57 .43•5 .64 59•5	13.5 18.5 12	27 34 25 Ø
PZX 72	8.0 Al 0.06 0 ₂ 0.02 W	As Rolled	67	63	10	20
PZX 63	5.2 Al 0.24 B 0.045 0 ₂	As Rolled	60.5	56.5	7.5	19.5
PZX 64	5.0 i.1 0.42 B 0.06 0 ₂	As Rolled	67 59•5	65	2 1.5	7.5 4.5
PZX 39	5 Al (N) 0.75 C (N) 0.11 N2	As Rolled Ann. 1000°C.	69	67 BRIT	-	-
PZX 40	5 Al (N) 1.5 C (N) 0.10 N2	Ann. 1000°C.		BRIT		
PZX 48	5.0 Al 5.04 Cr 0.065 02 0.14 N2	As Rolled As Rooled + 504 hrs. at 400°C. As Rolled + 504 hrs. at 500°C.	80.5	75.5 BRITI		12.5 ø
PZX 47	5.0 Al 12.03 Cr 0.105 02 0.09 N2	As Rolled Ann. 1000°C. Ann. 1000°C. + 24 hrs. at 400°C. Ann. 1000°C. + 504 hrs. at 400°C. Ann. 1000°C. + 504 hrs. at 500°C.	63.5	61.5 BRITT	7 LE	16 ø

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TABLE II (continued)

			No.			
B.N.F. Mark	Composition %	Condition#	U.T.S. tons/ sq.in.	tons/	Elonga- tion % on 1 in.	Redn. of Area
PZX 49	5 Al (N) 1 Cb (N) 0.115 02 0.08 N2	As Rolled + 504 hrs. at 400°C. As Rolled + 504 hrs. at 500°C.	53 57 55	49 55•5 52•5	16 11 5	45 29.5 ⁶ 16 8
PZX 50	5 Al (N) 5 Cb (N) 0.113 02 0.11 N2	As Rolled As Rolled + 504 hrs. at 400°C.	69 72	61.5 70	7 5	16 13 Ø
PZX 51	5 Al (N) 10 Cb (N) 0.093 02 0.11 N2	As Rolled + 504 hrs. at 400°C.	71.5 82	66 78	7 5	16 23 ø
PZ X 52	4.4 Al 1.27 Fe 0.095 02 0.13 N2 0.022 W	As Rolled + 504 hrs. at 400°C. As Rolled + 504 hrs. at 500°C.	67 73 73•5	61 70•5 70•5	9 6	25 20.5 12
PZX 93	5 Al (N) 3 Fe (N)	As Rolled	87	84	6	21.5
PZX 70	4.9 Al 5.0 Fe 0.069 02	As Rolled Ann. 1000°C. As Rolled + 504 hrs. at 400°C. As Rolled + 504 hrs. at 500°C.	98 74	93 72 BRIT		7.5 5
PZX 36	4.6 Al 1.5 Mo 0.136 02 0.06 N2 0.052 W	As Rolled Ann. 1000°C. As Rolled + 504 hrs. at 400°C. As Rolled + 504 hrs. at 500°C.	59 53 66•5 66•5	55.5 48.5 63.5	7 11.5 8	7.5 21 21.5¢
PZX 57	5.0 Al 4.9 Mo 0.11 02 0.12 N2 0.23 W	As Rolled Ann. 1000°C. Annealed + 504 hrs. at 400°C. Annealed + 504 hrs. at 500°C.	81 62.5 74.5 72.5	70.5 60 70.5	4 13.5 9	7.5 43 17.5 13 \$
PZX 91	8 Al (N) 5 Mo (N)	As Rolled Ann. 1000°C.	89.5 73.5	84.5 71.0	3.5 14.5	7•5 27•5

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TABLE II (continued)

beneditive resourcing, were product this decode will						
B.N.F. Mark	Composition %	Condition#	U.T.S. tons/ sq.in.	L of P tons/ sq.in.	Elonga- tion % on 1 in.	Redn. of Area %
PZX 58	4.9 Al 10.0 Mo 0.11 02 0.11 N2	As Rolled Ann. 1000°C. Ann. + 504 hrs. at 400°C. Ann. + 504 hrs. at 500°C.	67 84 79•5	- BRITTLE 63.5 79 77.5	10•5 7	23 15 Ø 12.5Ø
PZX 73	5.6 Al l Mn (N) 0.01 02	As Rolled	65 .	.61	7.5	26
PZX 74	5.9 Al 5 Mn 0.007 02 0.04 W	As Rolled	86	84	7	14.5
PZX 59	4.9 Al 0.52 Si 0.11 02 0.11 N2 0.018 W	As Rolled + 504 hrs. at 400°C. As Rolled + 504 hrs. at 500°C.	65•5 69•5 67	60.5 65 64.5	13.5 9	32 18•5¢
PZX 90	8 Al (N) 0.5 Si (N)	As Rolled		BRITTL BRITTL	1	
PZX 44+	5 Al (N) 1 Si (N) 0.10 N ₂	As Rolled As Rolled + 504 hrs. at 400°C. As Rolled + 504 hrs. at 500°C.	70 75 74	64 73 69	10 9 7	24.5 18.5 12.5
PZX 45	5 Al (N) 2 Si (N) 0.09 N2	As Rolled Ann. 1000°C.	73 57	66	3.5 2	6 5
PZX 68	5.2 Al 0.22 V 0.21 02	As Rolled	74•5	69.5	6.5	10.5
PZX 69	5.0 Al 3.9 V 0.36 0 ₂	As Rolled Ann. 1000°C.		BRITTI		
PZX 71	4.6 Al 8.7 V 0.17 O ₂	As Rolled Ann. 1000°C.	70	BRITTI 65	由 11.5	20.5
PZX 65	5 Al (N) 1 W (N) 0.05 02	As Rolled + 504 hrs. at 400°C.	57•5 66	51 64	5 4	20.5 17.5 ^ø
PZX 66	5 Al (N) 3 W (N) 0.10 02	As Rolled As Rolled + 504 hrs. at 400°C. As Rolled + 504 hrs. at 500°C.	70.5 77.5 76.5	64 74•5 72•5	6.5 8	15 23 ¢ 10.5¢
PZX 67	5 Al (N) 5 W (N) 0.07 O ₂	As Rolled + 504 hrs. at 400°C.	68.5 76.5	63	8 1	18 3.5%

TABLE II (continued)

B.N.F. Mark	Composition %	Condition ³⁴	U.T.S. tons/ sq.in.	L of P tons/sq.in.	Elonga- tion % on 1 in.	Redn. of Area
PZX 60	5 Al (N) 0.95 Zr 0.083 0 ₂	As Rolled + 504 hrs. at 400°C. As Rolled + 504 hrs. at 500°C.	63•5 69 65	59 68•5 63	12.5 6 5	17 9.5 ^ø 18.5 ^ø
PZX 6	5 Al (N) 5.1 Zr 0.067 0 ₂	As Rolled As Rolled + 504 hrs. at 400°C. As Rolled + 504 hrs. at 500°C.	68.5 72.5 70.5	. 62 ₄ 71 68	7 10	22.5 26.5%
PZX 62	5 Al (N) 9.5 Zr 0.05 O2	As Rolled As Rolled + 504 hrs. at 400°C. As Rolled + 504 hrs. at 500°C.	74 75•5 72•5	67.5 74 70.5	9 8 1	14.5 18.5 2 ø

Single specimen only, which was previously used for stress rupture test.

TABLE III
Stress Rupture Properties of Arc Melted Titanium

B.N.F. Mark	Composition	Testing Tempera- ture C.	Stress tons/ sq.in.	Life hours	Elong- ation % on 1 in.	Material Condition
PZY23 /7 /1 /3 /2	Ti 0.94 0.08 N	2 400	10 12 15 17 20	504# 476 32 0	2 31 29 18 20	Ann. 1000°C.
PZY31/7 /9 /1 /2	Ti 0.116 0, 0.08 N		8 10 12 15	606** 606** 288 0	1 1 35 21	As Rolled
PZY23 /4 /5 /6	Ti 0.094 0 0.08 N		3 5 7 9	504# 90 16 6	8 34 51 35	Ann. 1000°C.
PZY31/20 /11 /3 /4 /17 /21	Ti 0.116 0.08 N		2 3 4 5 6 7	604 ^H 504 ^H 295 67 78 1	. 1 12 65 79 76 71	As Rolled

^{*}Test discontinued.

⁽N) Nominal composition.

^{*}Specimens tested after 504 hrs. at 400° or 500°C. were unbroken stress-rupture test pieces which had extended less than 1%.

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TABLE IV

Stress Rupture Properties of Arc-Melted Titanium Alloys at 400 C.

B.N.F. Mark	Composition %	Stress tons/ sq.in.	Life Hours	Elong- ation % on 1 in.	Material Condition	Av. Grain Size mm.
PZX46/4 /6 /8	5 Al (N) 0.08 N ₂	30 35 37	504 [™] 273 12	<1 18 12	As Rolled	0.035
PZX72/5 /6 /1	8.0 11	35 37 38	504 ^H 504 ^H 504 ^H	1 2 - 3.	As Rolled	0.004
PZX63/8	5.2 Al 0.25 B (N)	35	390 - 456	14	As Rolled	0,017
PZX48/3 /15.	5.0 Al 5.04 Cr 0.105 0 ₂ 0.14 N ₂	30 35 38	504 ¹⁴ 504 ¹⁴ 504 ¹⁴	2 3 4	As Rolled	ø
PZX47/5 /8 /10 /11	5.0 Al 12.03 Cr 0.065 02 0.09 N2	25 Britt			Ann. 1000°C. side gauge l n of load	
PZX49/3 /13 /5	5 Al (N) 1 Cb (N) 0.115 0 ₂ 0.08 N ₂	25 30 35	504 ™ 0 0	1 15 20	As Rolled	0.028
PZX50/4 /6 /7	5 Al (N) 5 Cb (N) 0.113 0 ₂ 0.11 N ₂	30 35 38	504 [*] 504 [*] 7 min.	2 3 14	As Rolled	0,005
PZX51/3 /6	5 Al (N) 10 Cb (N) 0.093 0 ₂ 0.11 N ₂	30 35	504 ¹⁶	2 9	As Rolled	0.004
PZX52/5 /17	4.4 Al 1.27 Fe 0.13 N ₂ 0.022 W	30 35	504 ^元 504 ^元	2 11	As Rolled	0.007
PZX36/15 /4 /10 /12	4.6 Al 1.5 Mo 0.136 0 ₂ 0.06 N ₂ 0.052 W	20 25 30 35	504 ^H 504 ^H 504 ^H 393	<1 <1 2 16	As Rolled	0.01
PZX57/5 /10 /14	5.0 Al 4.9 Mo 0.12 N ₂ 0.23 W	30 38 40	504 ^M 504 ^M 504 ^M	<1 1 12	Ann. 1000°C.	0.003
PZX58/9 /7 /14	4.9 Al 10.0 Mo 0.11 N ₂	30 38 40	504 504 504 F	<1 1 2	Ann. 1000°.C.	0.002
PZX73/1	5.6 Al 1 Mn (N)	35	504 [™]	10	As Rolled	0.005
PZX74/2	5.9 Al 5 Mm (N)	37	504 ¹⁶	4	As Rolled	0.098
PZX59/11 /5 /8 /7 /10	4.9 Al 0.52 Si 0.11 N ₂ 0.018 W	25 30 35 38 40	50小 ^班 50小 ^班 50小 ^班 50小 ^班 50小 ^班	<1 .1 .2 4	As Rolled	ø

TABLE IV (continued)

B.N.F. Mark	Composition % Ti +	Stress tons/ sq.in.	Life Hours	Elong- ation % on 1 in.	Material Condition	Av. Grain Size mm.
PZX44/3 /11 /9 /13	5 Al (N) 1 Si (N) 0.10 N ₂	25 35 38 40	504 [±] 504 [±] 504 [±] 504 [±]	<1 2 <1 2	As Rolled	ø
PZX71	4.6 Al 10 V (N)	35	504 ^{**}	2	Ann. 1000°C.	0,098
PZX65/17 /5	5 Al (N) 1 W (N)	32 35	504 ³⁴ 363	2	As Rolled	0,003
PZX66/2	5 Al (N) 3 W (N)	35 -38	504 ^{**} 504 ^{**}	<1 4	As Rolled	0.003
PZX67/7 /13	5 Al (N) 5 W (N)	35 40	504 [™] 504 [™]	<1 11 .	As Rolled	0,002
PZX60/9	5 Al (N) 0.92 Zr	35 40	504 ^H	<1 :	As Rolled	0.029
PZX61/3	5 Al (N) 5.1 Zr	35 40	504 [±] 504 [±]	1 2	As Rolled	0,025
PZX62/3 /11	5 Al (N) 9.5 Zr	35 40	504 [#] 504 [#]	1 .	As Rolled	0.014

Test discontinued

(N) Nominal composition

Grain size not clear in 'as rolled' condition

Stress Rupture Properties of Arc-Melted Titanium
Alloys at 500°C.

B.N.F. Mark	Composition % Ti +	Stress tons/ sq.in.	Life Hours	Elong- ation % on 1 in.	· Material Condition	Av. Grain Size mm.
PZX46/9 /5 /16 /15	5 Al (N) 0.08 N ₂	12 14 15 16	504 [¥] 349 241 117	17 67 49 23	As Rolled	0.035
PZX72/8 /3	8.0 Al	14 16	504 ¹ 504 ¹	1 3	As Rolled	0,004
PZX63/0	5.2 Al 0.25 B (N)	14	321	38	As Rolled	0.017
PZX48/4 /16 /12	5.0 Al 5.04 Cr 0.065 02 0.14 N2	14 16 17	504 [¥] 504 [¥] 504 [¥]	2 4 9	.As Rolled	ø

/cont'd

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TABLE V (continued)

B.N.F. Mark	Composition % Ti +	Stress tons/ sq.in.	Life Hours	Elong- ation % on 1 in.	Material Condition	Av. Grain Size
PZX47/6	5.0 Al 12.03 Cr 0.105 02 0.09 N2	12.	.504 [#]	1	Ann. 1000°C.	0.034
PZX49/4 /11	5 Al (N) 1 Cb (N) 0.115 0 ₂ 0.089 N ₂	12 14	504** 151	14 56	As Rolled	0.028
PZX50/11 /5 /16	5 Al (N) 5 Cb (N) 0.113 02 0.11 N2	12 14 16	504 [#] 371 218	29 62 36	As Rolled	0.005
PZX51/10 /15	5 Al (N) 10 Cb (N) 0.093 0 ₂ 0.11 N ₂	12 16	504 [#] 316	13. 45	As Rolled	0.004
PZX52/11 /14	4.4 Al 1.27 Fe 0.13 N ₂ 0.022 W	14 16	· 504** 37	3 27	As Rolled	0.007
PZX36/5 /6 /11 /17	4.6 Al 1.5 Mo 0.136 02 0.06 N2 0.052 W	10 12 14 16	504 ^x 504 ^x 504 ^x 504 ^x	1 2 4 18	As Rolled	0,01
PZX57/12 /7 /6	5.0 Al 4.9 Mo 0.12 N ₂ 0.23 W	14 16 18	504 [±] 504 [±] 504 [±]	<1 <1 1	Ann. 1000°C.	0,003
PZX58/12 /6 /10	4.9 Al 10.0 Mo 0.11 N ₂	14 15 16	504 ^x 504 ^x 504 ^x	7 2 3	Ann. 1000°C.	0.002
PZX73/1 /0	5.6 Al 1 Mm (N)	14 16	154 58	58 - 44	As Rolled	0.005
PZX74/7	5.9 Al 5 Mm (N)	16	160	40	As Rolled	0.098
PZX59/4 /13 /15	4.9 Al 0.52 Si 0.11 N2 0.018 W	12 14 16	504 ³⁶ 504 ³⁶ 504 ³⁶	<1 · 2 · 3	As Rolled	
PZX44/4 /10 /6 /5	5 Al (N) 1 Si (N) 0.10 N ₂	14 15 16 17	504. 504. 504. 504. 504. 504. 504. 504.	<1 3 3 8	As Rolled	
PZX68/8	5.2 Al 1 V (N)	14	360	16	As Rolled	0.004
PZX71	4.6 Al 10 V (N)				Ann. 1000°C.	0.098
PZX65/4	5 Al (N) 1 W (N)	14	280	29	As Rolled	0,003
PZX66/3	5 Al (N) 3 W (N) 5 Al (N) 5 W (N)	14	504 ³⁶	3	As Rolled	0.003
/3	(LT) 2, 11 (LT)	16	504	* 3		
PZX60/15	5 Al (N) 0.92 Zr	14	504#	4	As Rolled	0.029
PZX61/14	5 Al (N) 5.1 Zr	14	504.35	2	As Rolled	0.025
PZX62/13/2	5 Al (N) 9.5 Zr	14 16	504 [™] 504 [™]	2	As Rolled	0.014

*Test discontinued

(N) Nominal Composition

pGrain size not clear in 'as rolled' condition.

TABLE VI

Results of X-ray Examination of Titanium Alloys after Various Heat Treatments

tr. = trace s.a. = small amount P = unidentified phase Figures in brackets give estimated amount of phase.

TABLE VII

Hardness (V.P.N.) of Titanium and Titanium Alloys after

Various Heat Treatments

	2	Λs	Rolled Sheet	Sheet	SHEET		
B.N.F. Mark	Composition % (Nominal)	Melted Ingot		Annealed 1000°C.	₩. Q. 600°C.	W. Q. 800°C.	W. Q. 1000°C.
PZY 23 31 32	Ti (Sponge QAB) Ti " Ti "	182 192 187 (182)	199 179 190	163			219
35 PZX 46 72 59 44 45 47 48 49 50 51 36 57 58 52 70 60 61 62 65 66 67 63 64 68 69 71 73	5 Al 0.5 Si 5 Al 0.5 Si 5 Al 1.0 Si 5 Al 2.0 Si 5 Al 12 Cr 5 Al 10 Cb 5 Al 10 Mo 5 Al 10 Mo 5 Al 10 Fe 5 Al 10 Fe 5 Al 10 Fe 5 Al 10 W 5 Al 3 W 5 Al 5 Al 10 S 5 A	311 306 334 351 384 345 321 293 347 329 347 329 340 377 381 396 343 340 371 500 364 290 348	312 346 314 354 354 368 319 348 320 338 352 339 430 335 357 357 363 407 341 407 341 420	292 315 306 313 343 381 350 281 304 328 317 345 324 338 358 281 304 335 264 353 369 299 371 385 349 280 338	288 294 305 304 315 498 360 269 295 286 309 334 365 346 376 292 351 280 322 351 280 322 270 309 344 417 343 310 338	289 288 305 314 335 377 347 266 318 301 289 320 335 335 365 289 306 324 306 339 310 288 311 344 369 326 276 327	264 309 315 328 321 341 323 272 325 362 344 318 293 352 302 321 363 308 318 352 309 352 421 393 366

*Annealed ingot

TABLE VIII

Effect of Heat Treatment on the Hardness of Some Titanium Alloys

		Hardness V.P.N.				
B.N.F.	Composition % (Nominal)	Annealed 1000°C.	Quenched 1000°C.	Quenched 1000°C. Aged 1 hr. at 400°C.	Quenched 1000°C. Aged 15 days at 400°C.	
PZY 31 PZX 46 63 51 44 67 36 62	Ti Ti 5 Al Ti 5 Al Ti 5 Al 10 Cb Ti 5 Al 1 Si Ti 5 Al 1 Mo Ti 5 Al 5 W Ti 5 Al 10 Zr	163 292 299 328 313 317 309 335	219 264 309 362 328 344 318 363	225 300 304 342 320 324 335 370	210 309 325 370 324 340 362 374	

APPENDIX

Metallographic Observations on Titanium and Its Alloys

All the alloys described in the main part of the report were examined micrographically in all conditions; most were also submitted to X-ray examination. As the metallographic work was only incidental to the main purpose of the research, no attempt was made to explore the alloy systems thoroughly, but it is nevertheless thought worth while to record some of the observations made and the conclusions drawn.

Metallographic Techniques

Both electrolytic and mechanical polishing have been used.

Jacquet's (10) reagent proved fairly successful for most alloys, but difficulty was experienced with alloys showing clear equiaxial alpha structures; these tended to develop undulating surfaces which did not prohibit micro-examination but made photography unsatisfactory. The greatest disadvantage of this method, however, is that specimens cannot be polished after mounting in bakelite. Since one bakelite mount can easily accommodate 10 or 11 samples of sheet 0.060 in. thick, it was found more convenient to mount the samples thus and polish mechanically.

Mechanical polishing was on Selvyt cloth impregnated with diamond particles of less than 1 micron diameter. It was necessary to grind to 6/0 emery before polishing with diamond, the "Bluebell" stage being omitted. The Selvyt was moistened with white spirit and polishing took about three hours.

Various mixtures of hydrofluoric and nitric acids in water were used for etching; the proportions are by no means critical. The plain equiaxial alpha structures, which were always difficult to prepare, were sometimes much improved by swabbing with 50% nitric acid.

A thermal etch is readily developed on titanium alloys and samples annealed in vacuo normally showed a bright macro-etched surface. A rather different example, showing the underface of an ingot after the top had been molten in the arc furnace, is illustrated in Plate VIA.

In all photographs of wrought materials the rolling direction is parallel to the long edge of the photograph.

Interpretation of Structures

This is discussed in the main report.

Structures found in Ternary Alloys

The titanium/aluminium diagram due to Bumps, Kessler and Hansen (2) shows that at 5% aluminium the alpha+beta field extends from 955°C. to 975°C. The alpha/alpha+beta transus line rises to 1005°C. at 8% aluminium and 1020°C. at 10% aluminium.

All the third elements, except carbon, which were added to titanium containing 5% of aluminium lower the transformation temperature of pure titanium; therefore, for all the ternary alloys studied, the transformation temperature should be lower than 975 C. Accordingly, heat treatment at 1000°C. followed by quenching should produce structures containing either alpha and beta or only beta, where the beta-stabiliser's influence is sufficiently strong. Where the influence of the aluminium predominates, alpha stable at the quenching temperature or alpha formed from the beta during the quench, or both, should be present.

Alloys consisting entirely of alpha solid solution at room temperature were obtained when 1% of columbium or up to 9.5% of zirconium were added to the 5% aluminium alloy regardless of heat treatment. The silicon

(up to %), carbon (up to 1.5%) and boron (up to 0.5%) alloys consisted only of alpha solid solution and widely dispersed particles of intermetallic compounds. The compounds found in the alloys containing silicon and carbon had the structures associated with the formulae Ti5Si3(12) and TiC. The compound occurring in the boron-bearing alloys is unidentified. In the boron-bearing alloys the particles lay in lines parallel to the rolling direction (Plate VIB) and both in these alloys and in those containing silicon were smaller than in the carbon-bearing alloys; the latter exhibited an interesting phenomenon which will be discussed more fully below.

All these alpha alloys, whether containing primary particles or not, were finer grained than the corresponding binary titanium/aluminium alloys.

Only one alloy consisted entirely of beta in any of the conditions studied. This was titanium containing 5% of aluminium and 12% of chromium, as rolled or quenched from 1000°C. All the remaining alloys, after annealing or quenching from 800°C. or 600°C. consisted either of alpha only or of alpha and beta in varying proportions according to heat treatment; alloys containing 1% iron, 3.9% and 8.7% vanadium, 1% or 5% manganese, and 5% of molybdenum behaved as for composition m in Fig. 2.

Structural Changes on Ageing

Only the alloys containing 5 and 12% of chromium, 5% of manganese, 5% of iron and 8.7% of vanadium show significant hardening on ageing at 400°C. or 500°C. Of these, the 12% chromium alloy and the 5% iron alloy showed no structural changes on ageing. The 5% chromium and 5% manganese alloys were very similar in structure after quenching, consisting of small clear particles of alpha in a transformed matrix; after ageing the small clear particles had been partly absorbed. The structure of the 10% vanadium alloy appeared to contain more alpha after ageing than after quenching from 1000°C.

This completes the general survey of the structures of these alloys, but one or two points of interest which arose during the work are considered worth further discussion.

Embrittling Compound in Titanium/Aluminium/Carbon Alloys

Certain titanium/aluminium alloys, after heat-treatments at temperatures between 500°C. and 1000°C., were observed to contain a finely-divided precipitated phase. The distribution and amount of the precipitate depended on the aluminium content, the purity of the alloy, the temperature of heat-treatment and the time at that temperature. The effect of the precipitation (in the 1, aluminium alloy at least) was to reduce the elongation of the materials, very markedly in the carbon contaminated krollmelted alloys, but to a much lesser extent in the arc-melted materials. The tensile strength and hardness were little affected. The observations leading to the above conclusions are described below.

Materials

The materials used fall into three groups; (i) those melted in a Kroll type of graphite resistor furnace(4) (nominal aluminium contents of 1%, 2%, 3%, 4%, 6% and 8%), (ii) those melted in the arc furnace (nominal aluminium contents of 1%, 5% and 10%) containing no added carbon and (iii) arc-melted alloys containing 5% of aluminium with 0.75% and 1.5% nominal carbon content. Kroll-melted alloys normally contain more than 0.4% of carbon; the arc-melted material should contain only about 0.05% carbon or less unless carbon was deliberately added. A list of alloys examined is given in Table I, below.

TABLE I
Composition of Materials

B.N.F. Mark	Melting Method	Nominal Aluminium Content	Approx. Carbon Content	Oxygen Content (Anal.)	Nitrogen Content (Anal.)
OGZ 4 OGZ 31 OGZ 58 OGZ 30 OGZ 63 OGZ 64	Kroll (Graphite crucible) " " " "	1 (anal.) 2 3 4 6 8	0.65(anal.)	0.111	
PZX 13 PZX 1 PZX 2	Arc "	1 4.22(anal.) 7.48(anal.)	0.05	0.076	
PZX 39 PZX 40	11	5 5	0.75		0.11 0.10

(anal.) = by analysis

Occurrence of the Precipitate

In Kroll-melted alloys the precipitate has only been observed within the grains of the specimens containing 4% or less of aluminium. Heattreatment at 900°C. produced no precipitate in the alloys containing 1% or 4% of aluminium but it was present, in small quantities, at 2% and 3% of aluminium. After heat-treatment at 800°C., 700°C. or 600°C. (5 hours) the alloys containing 1, 2, 3 or 4% of aluminium all showed the precipitate. In the 1% aluminium alloy, after heating at 600°C., it was very dense, and distributed at random. (Plate VIIA). A highly ordered distribution was produced by heating the 4% alloy at 800°C. for 2 hours (Plate VIIB) and almost any density of distribution and degree of order between these two occurred according to temperature and composition; another example is illustrated by Plate VIIC. Plate VIID shows the appearance of the particles at high magnification. A little precipitate, similar in distribution to that in Plate VIIA, but very much less in quantity, was produced in the 1% aluminium alloy after 5 hours at 500°C., but shorter times at this temperature or 5 hours at lower temperatures failed to produce it in any of the materials.

Four materials prepared by arc melting (but without deliberate carbon additions), unalloyed titanium and alloys containing 1%, 4% and 7.5% of aluminium, were examined after two treatments only; (i) the anneal at 1000°C. followed by slow cooling and (ii) treatment (i) followed by 5 hours at 600°C. and water quenching. The latter treatment, which was known to produce the heaviest precipitation in the Kroll melted 1% aluminium alloy, caused traces of the precipitate to appear in the alloy containing 1% of aluminium, but it was not seen in any of the other materials.

The precipitate did not appear in any of the ternary alloys made in the arc furnace except those to which carbon had been added deliberately. Two such alloys, containing 5% of aluminium and 0.75% or 1.5% of carbon, were examined. In the as-rolled condition they contained a heavy precipitate randomly distributed as in Plate VIIA. After quenching from 800 C. the precipitate appeared in an ordered array. It was not seen after other heat-treatments.

Electron micrographs of the Kroll melted 3% and 4% aluminium alloys revealed that the particles were discs, or possibly needles, set at an angle and lying in piles along planes in the grains (Plate VIIE). This is clearly shown by the fact that the particles on any one plane throw shadows all in one direction, which differs, however, from one plane to another (Plate VIIF). Many boundaries were found to consist of similar particles closely packed which had even in some places, merged; others, however, remained clear and free from precipitate.

Effect of the Precipitate on Mechanical Properties

Only a very small quantity of material being available, mechanical tests were of necessity very limited in number, but the results give some indication of the effect of the precipitation, particularly on elongation. The materials chosen were Kroll melted and are melted 1% aluminium alloys, and they were tested in two conditions, (i) after annealing for 1 hour at 1000°C. in vacuo and cooling slowly in the furnace, and (ii) after treatment (i) followed by holding for 5 hours at 600°C. in argon and quenching ment (i) followed by holding for 5 hours at 600°C. in argon and quenching in water; the latter treatment was known to produce very heavy precipitation in Kroll-melted material. The Hounsfield tensometer tensile test was used, the specimens being \(\frac{1}{4} \) in. in width over a 1 in. gauge length. The Kroll melted material was 0.060 in. thick and the arc-melted material 0.030 in. thick. The results are given in Table II below.

TABLE II

Mechanical Properties of Titanium Containing 1% Aluminium

B.N.F. Mark	Melting Method	Condition	U.T.S. tons/sq. in.	Elonga- tion %	Redn. Area	Hard- ness V.P.N.
OGZ 4	Kroll	Ann. 1000°C. Ann. 1000 C. + 5 hr. 600°C. water quench	58.9 (mean of 3) 53.5 (1 only)	18	38 3•5	295 301
PZX 13	Arc	Ann. 1000°C. Ann. 1000°C. + 5 hr. 600°C. water quench	24.5 (mean of 2) 24.3 (mean of 3)	43 33•5	37 28	138 139

It can be seen that, whereas the strength and hardness of both materials were not much affected by the low temperature heat-treatment, the elongation of the Kroll melted alloy was drastically reduced. This effect was reproduced but to a much lesser extent, in the arc melted alloy.

The Identity of the Precipitated Phase

Only one experimental method has been used in attempts to identify the precipitate.

The method used was to over-etch grossly, so that precipitated phases stood proud of the matrix, and examine the surface by electron diffraction. A sample of the Kroll-melted 3% aluminium alloy, which contained a heavy precipitate, was examined in this way. A complete pattern of titanium carbide was obtained and titanium lines were absent, indicating that the technique had been successful, but giving no information about the precipitate, as large particles of carbide are present in all Kroll-melted alloys. A similar technique applied to are-melted materials gave only a titanium pattern.

The main differences in composition between arc-melted alloys and Kroll-melted alloys made from the same supplies of metal is in carbon content. There may also be differences in oxygen, nitrogen or hydrogen content, or in more than one of these, resulting from the different melting techniques in the two furnaces. The precipitate, therefore, may contain aluminium and one or more of these elements; alternatively, it need not necessarily contain any of them, but may be thrown down as the result of changes in the oxygen, nitrogen or hydrogen contents brought about hy heattreatment.

Hydrogen seems unlikely to be a constituent of the compound, as the samples containing the precipitate had all been annealed at 1000°C. in vacuo before the heat-treatments at lower temperatures (in purified argon) which caused the precipitation. It is known that all hydrogen is readily removed from titanium by vacuum heat-treatment at temperatures above 850°C. (13)

Diffusion of oxygen and nitrogen into titanium is extremely slow, and composition differences in the main body of the material are unlikely to result from the short heat-treatments given.

There remains carbon. In view of the appearance of the precipitate in quantity resulting from the deliberate addition of carbon to arc-melted materials, which otherwise showed only traces of it, it becomes almost certain that carbon causes the precipitation; it does not necessarily follow that carbon is a constituent of the precipitate, which remains unidentified.

Changes in Transformed Structures Occurring on Heating Below the Transformation Point.

In pure titanium, the alpha formed from beta when the metal passes through the transformation point differs only in structure from the beta. In the alloys, while the average composition remains unchanged, the first alpha to separate will have a different composition from the matrix beta, according to the usual rules, as well as a different structure. On subsequent annealing, therefore, diffusion may be expected to take place, with corresponding effect upon the microstructure. In worked material, recrystallisation may also occur. In connection with the impact tensile tests described in the main report, it became of interest to know whether structural changes were occurring in the alloys during heating in the furnace before breaking. A series of heat-treatments were therefore given to the binary alloys containing 5, 8 and 10% aluminium, and the effect on the microstructure was studied.

The rolled plates, $\frac{1}{4}$ in. thick, all had a transformed structure similar to that shown in Plate IIIA, indicating that cooling had been rapid across the alpha/alpha+beta transus temperature. After 5-10 minutes in the furnace before breaking and quenching at temperatures between 600°C. and the transformation point, these structures were found to persist; specimens broken and quenched above this temperature showed a transformed structure of more clearly defined platelets, Plate IIIB. Recrystallisation had not occurred in any specimen, although the final pass in rolling had produced 16% reduction in thickness and no subsequent anneal had been given.

The heat-treatments, which were carried out in air, showed that at all compositions the structure of the rolled material persisted for at least 16 hours at 600°C. The 5% aluminium alloy recrystallised completely after 2 hours at 900°C., five minutes at this temperature producing no effect and 15 minutes causing the onset of recrystallisation in some parts of the specimen. Even after recrystallisation, the original transformed structure of the rolled material could still be seen faintly in the background of the equiaxial grains, which were very much smaller than the grains of the rolled material (Plate IIIC). The specimens broken at 1000°C, had been in the alpha+beta region and had a typical transformed structure. The alloy containing 8% of aluminium showed similar charac-

teristics, recrystallisation beginning after 15 minutes at 900°C. but only being complete after 2 hours at 1000°C., the original transformed structure was again visible in the background. The specimen quenched from 1100°C. had been in the alpha-beta range.

The alloy containing 10% of aluminium had a rather different structure after rolling. The general appearance was of a transformed alloy, but each platelet was heavily veined with dark lines (Plate IIID). On heating for periods of up to 2 hours at 1000°C. or for longer times at lower temperatures, the veining faded slowly until an almost clear dendritic structure was obtained (Plate IIIE). After 10 minutes at 1100°C, the alloy had recrystallised to very large equiaxed grains, but had also been in the alphate tarange and the grains consisted of thick boundary layers of transformed material surrounding clear alpha areas (Plate IIIF).

It is clear from the foregoing that changes in the structures of these alloys do take place on annealing. If the original transformed structure represents concentration gradients these are only very slowly dispersed on annealing. Recrystallisation does occur, but, with the degree of residual work present in these specimens, temperatures of about 100°C. below the alpha/alpha+beta transus line are necessary before it can be initiated. Further evidence that recrystallisation is slow in starting and requires considerable heat even after heavy working was provided when 5% and 10% aluminium alloys, some of which had been cold-worked as much as 37%, were annealed to facilitate further rolling. Even with this high degree of cold work, a minimum temperature of 850°C. was found necessary to initiate recrystallisation.

SUMMARY

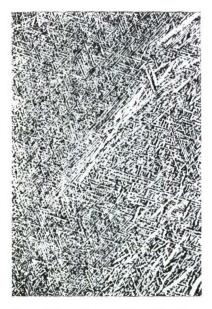
Binary alloys of titanium with aluminium and ternary alloys of these two elements with many others have been examined in various conditions. A great variety of structures has been found, all of which are consistent with the view that 5% of aluminium added to titanium does not materially alter the alloy systems formed between titanium and the third element. Ageing characteristics could not be directly linked with changes in microstructure.

An unidentified minor constituent has been observed in titanium/ aluminium/carbon alloys in certain conditions and its presence has been found to embrittle the alloys.

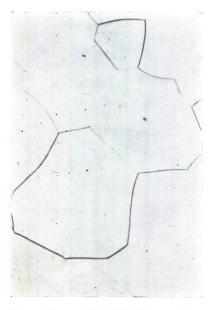
The effect of heat-treatment on transformed structures, produced by rapid cooling of wrought binary titanium/aluminium alloys, has been studied. It has been found that such structures can persist even after recrystallisation has occurred.

Plates I - VII and Figures 1-6 were provided by the British Non-Ferrous Metals Research Association.

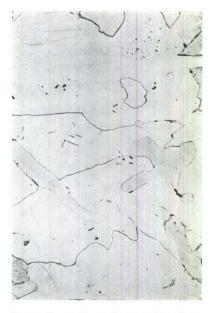
TYPES OF STRUCTURE FOUND IN TITANIUM ALLOYS



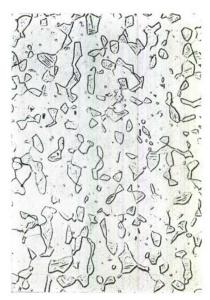
A.—a. Formed on rapid cooling from the β field. $\times200$



C.— α . Recrystallised in the α region. $\times 400$

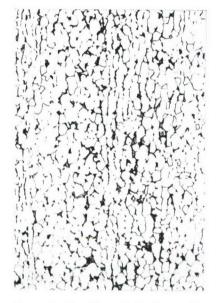


B.—a. Formed on slow cooling from the β field. $\times\,65$

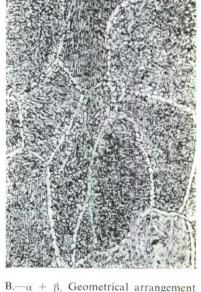


D.— α and acicular α . Quenched from the $\alpha+\beta$ field but β not retained. $\times\,200$

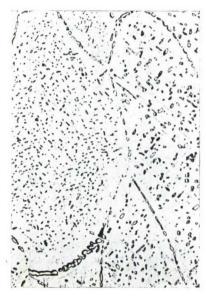
TYPES OF STRUCTURES FOUND IN TITANIUM ALLOYS



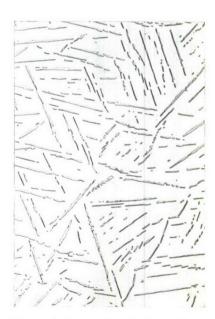
A.— α + β . Quenched from the α + β field. β retained on quenching. $\times 200$



B.— α + β . Geometrical arrangement of α and β platelets. \times 200

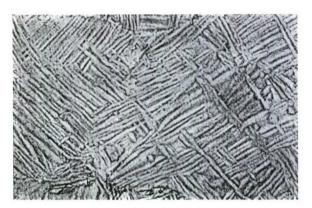


C.— α + β . α particles in α + β matrix and at grain boundaries. \times 200

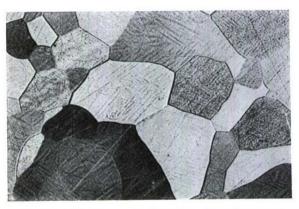


D.— α + β . α spines in β matrix. $\times 100$

CHANGES IN TRANSFERRED STRUCTURES ON ANNEALING



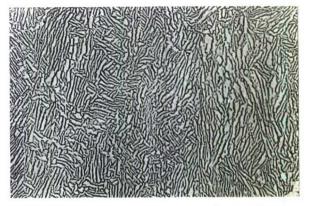
A.—Ti—5% Al. α —Transformed structure produced by quench after last pass through rolls. \times 100



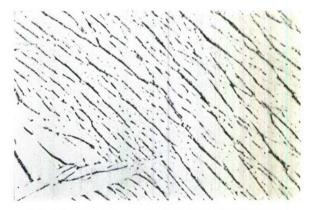
C.—Ti—8% Al. α —Rolled plate. Two hours at 1,000° C, and quenched. \times 100



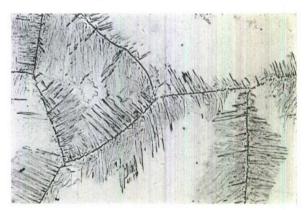
E.—Ti—10% Al. α —Rolled plate. Two hours at 1,000° C. and quenched.



B.—Ti—5 % Al. α —Impact tensile specimen broken at 1,000 ° C. and quenched. \times 100



D.—Ti—10% Al. α —Rolled plate. Two hours at 900° C. and quenched. \times 400



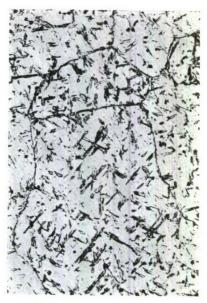
F.—Ti—10% Al. α and acicular α —Impact tensile specimen broken at 1,100° C. and quenched. β present at test temperature but not retained. \times 100



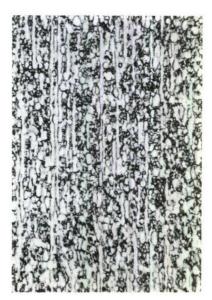
A.—Ti—5% Al—12% Cr. β —As rolled. \times 600



C.—Ti—5% Al—5% Mo. α + β — × 400

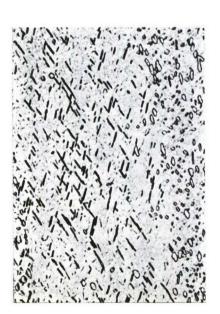


B.—Ti—5% Al—12% Cr. α in β matrix. Annealed 1,000° C. \times 200





A.—Ti—5% Al—8.7% V. α + β — \times 400



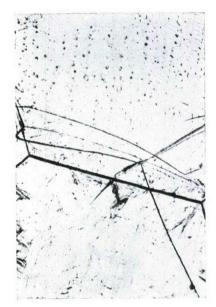
C.—Ti—5% Al—5% Cr. Mainly a. As rolled. $\times\,600$



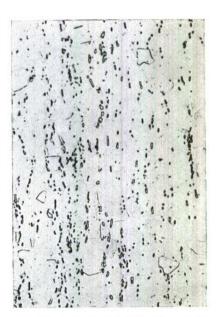
B.—Ti—5 % Al—8.7 % V. α + β — Annealed 1,000 $^{\circ}$ C. \times 200



D.—Ti—5 % Al—5 % Cr. Water quenched 1,000 $^{\circ}$ C. and aged 61 days at 500 $^{\circ}$ C.

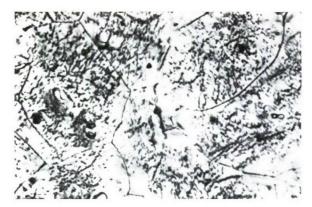


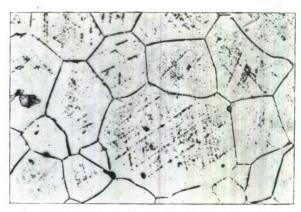
A.—Ti—10% Al. Surface of ingot, Thermal etch. $\times\,55$

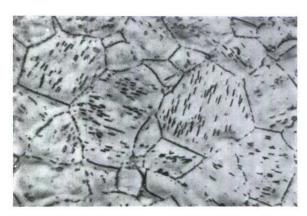


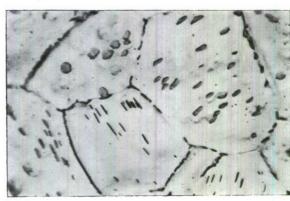
B.—Ti—5% Al—0.5% B. Quenched from 1,000° C. $\times\,200$

EMBRITTLING CONSTITUENT IN Ti Al-C ALLOYS





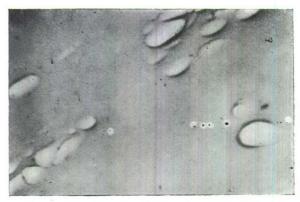




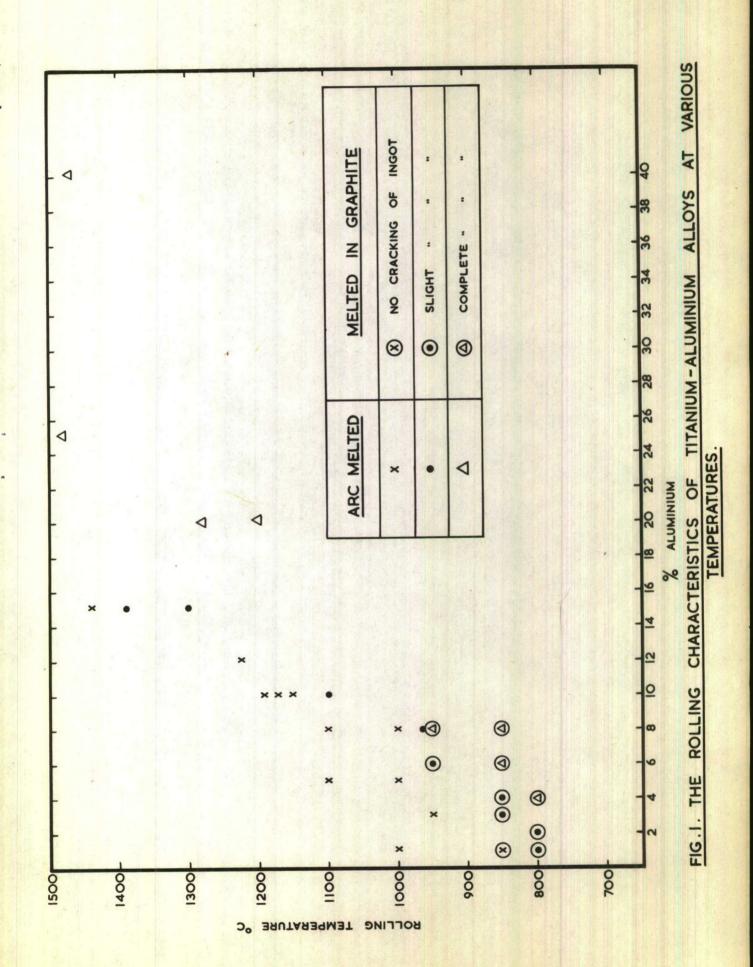
D.—As (C). ×1500



E.—Ti—3% Al. Electron micrograph of precipitate produced by heat treatment at 800° C. $\times\,6000$



F.—As (E). Showing shadows thrown in different directions. $\times\,8500$



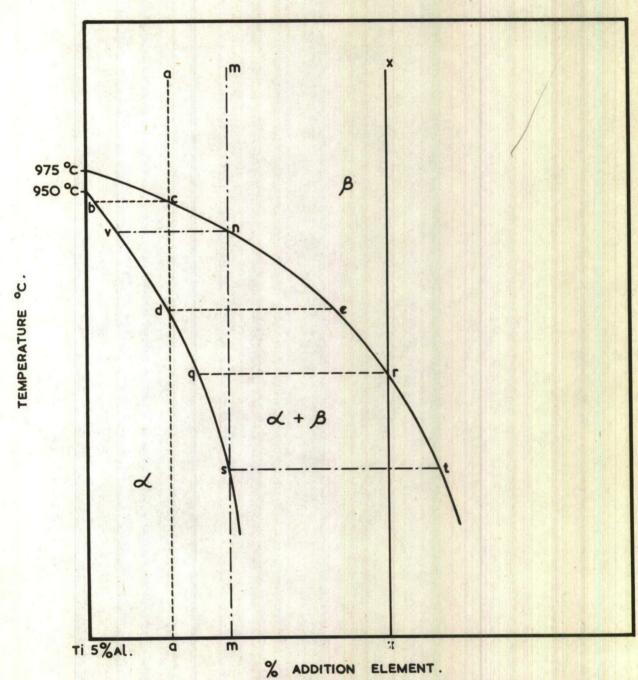


FIG. 2. PSEUDO BINARY EQUILIBRIUM DIAGRAM OF
TITANIUM 5% ALUMINIUM WITH ANOTHER
ELEMENT M.

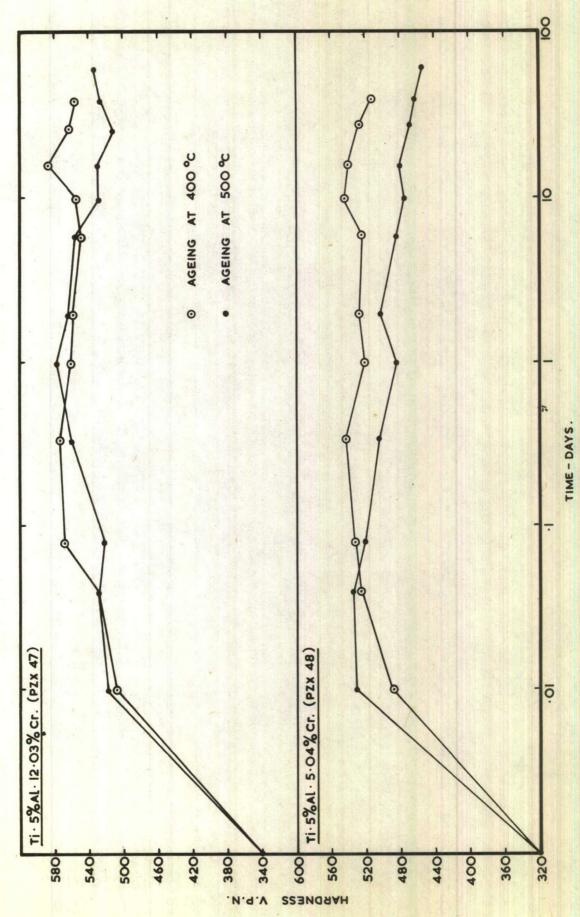


FIG. 3. AGEING CURVES FOR TI-AI-Cr ALLOYS.

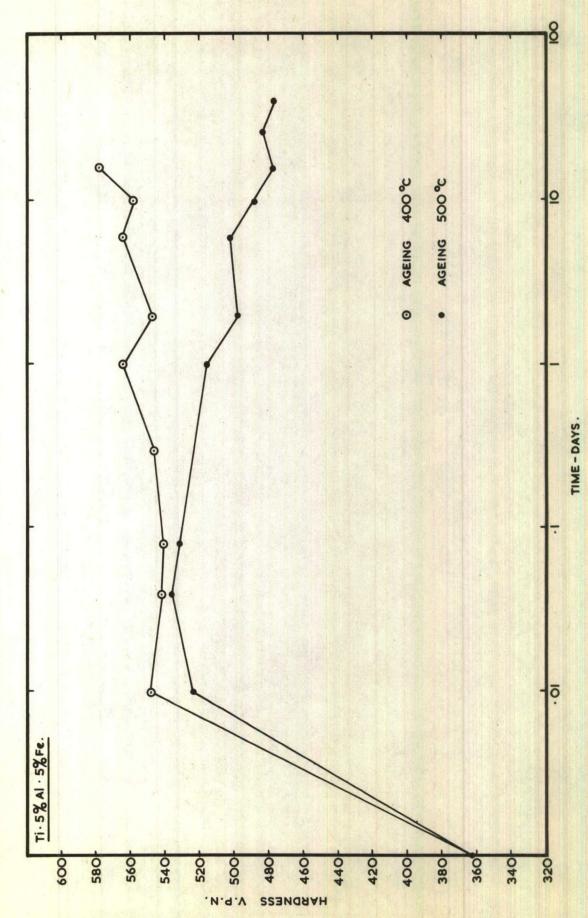


FIG. 4. AGEING CURVES FOR TI-AI-FE. ALLOY.

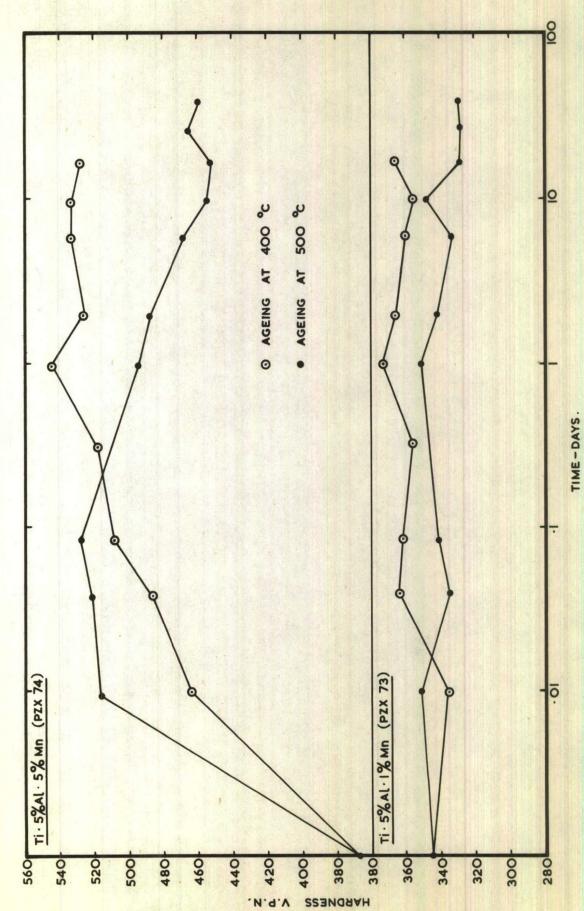
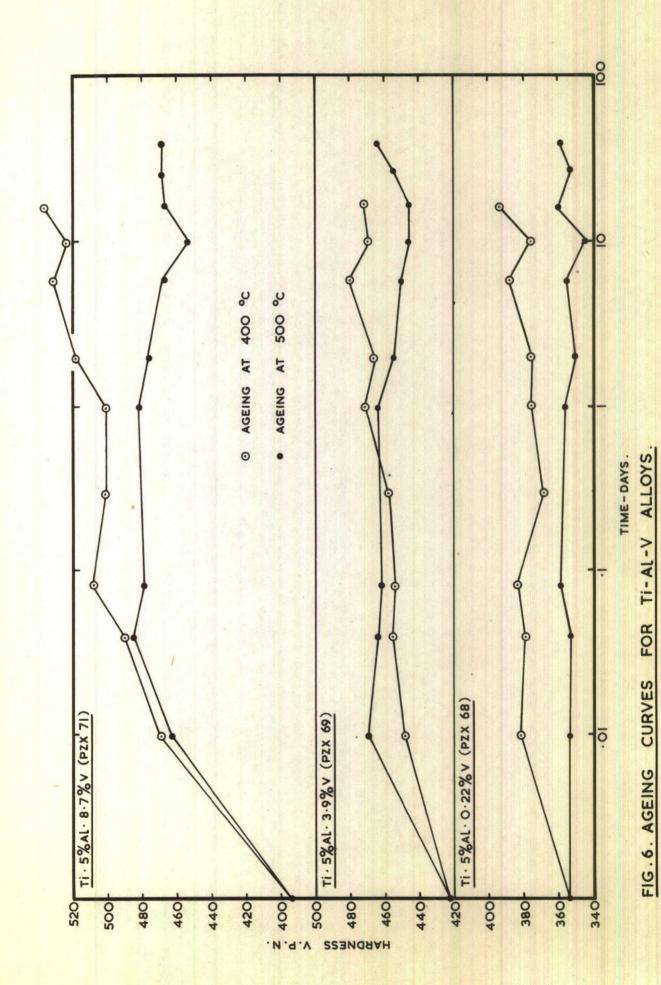


FIG. 5. AGEING CURVES FOR TI-AL-Mn ALLOYS.



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